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THOUGHTS ABOUT THE OPTIMUM DATA ACQUISITION GEOMETRYAND TIME RESOLUTION OF MONOCHROMATIC BEAM X-RAY DIFFRACTION MICROSCOPY EXPERIMENTS

Wolfgang Ludwig[1,2]
Peter Reischig[3,4], Andrew King[5], Michael Herbig[1],
Henry Proudhon[6] , J.Y. Buffiere[1], Simon Rutishauser[7], Christian David[7]

[1]MATEIS, INSA de Lyon, Villeurbanne, France,
[2]ESRF, Grenoble, France
[3]Karlruhe Institute of Technology, Germany
[4]Delft University of Technology, Delft, The Netherlands
[5]GKSS, Geesthacht, Germany
[7]Mines, Paristech, France
[8] Laboratory for Micro- and Nanotechnology, PSI, Switzerland

ABSTRACT

So far, 3D X-ray diffraction microscopy (3DXRD) and X-ray diffraction contrast tomography (DCT) experiments have typically been performed in forward scattering geometry, the detector intercepting diffraction cones up to limited opening angles below 30°. The extension of the current synchrotron–based methodology towards 3D orientation mapping of deformed microstructures with down to (sub-)micrometer spatial resolution will require a reduction of the pixel and hence sample size by one order of magnitude. This in turn relaxes the need for high energy beams and opens interesting possibilities for new acquisition geometries, taking advantage of improved spatial resolution and strain sensitivity at high diffraction angles. First results obtained in this acquisition geometry will be discussed and a comparison to polychromatic micro-diffraction experiments is drawn.

1. INTRODUCTION

The characterization of polycrystalline materials in terms of the local parameters of the crystalline lattice requires determination of nine parameters (three representing orientation and six for the elastic strain state) in three-dimensional space and time. There are two fundamental approaches for 3D mapping by means of synchrotron radiation: i) polychromatic micro-diffraction and its extension into 3D by differential aperture microscopy (DAXM) (Larson, Yang, Ice, Budai and Tischler 2002)and ii) monochromatic beam diffraction techniques like

3DXRD (Poulsen 2004) and DCT (Ludwig, Reischig, King, Herbig, Lauridsen, Johnson, Marrow and Buffiere 2009). Both have their respective strengths and limitations, which shall be briefly reviewed here.

1.1 Polychromatic micro-diffraction and DAXM Orientation mapping by means of polychromatic micro-diffraction resembles conceptually electron backscatter diffraction (EBSD) in the scanning electron microscope: the sample (or the micro-probe) are scanned over the area of interest and for each position a diffraction pattern is collected on a low resolution 2D detector screen. The detector is typically positioned with its surface normal perpendicular to the beam and intercepts diffracted beams up to ±45°. Due to the high penetration of X-rays, information is collected from an extended volume (typically several tens up to several hundreds of microns, depending on the X-ray spectrum and attenuation of the material) and the diffraction patterns of all grains in this interaction volume will overlap on the detector. Scanning an absorbing wire across the sample surface and analysing the difference between consecutive image frames, one can isolate the contributions from different positions in the 3D interaction volume. This is the principle of differential aperture microscopy (DAXM) (Larson et al. 2002), providing access to the local orientation and deviatoric part of the elastic strain tensor at each point in the 3D sample. Due to the isolation of diffraction signals from small sample volumes, DAXM can more easily accommodate deformed and/or multiphase materials. The ultimate spatial resolution is given by the size of the focal spot. State of the art achromatic (reflective) focussing optics at 3rd generation synchrotron sources provide sub-micron resolution as a matter of routine and spot sizes below 100 nm have already been reported (Liu, Ice, Tischler, Khounsary, Liu, Assoufid, and Macrander 2005).

The principal limitation of DAXM is the poor time resolution implied by the 3D scanning procedure. Assuming a scanning frequency of 10 Hertz, the characterization of a moderate sample volume of 100x100x100 voxels requires already more than a full day. DAXM provides therefore only limited capabilities for the observation of microstructure *evolution* during in-situ testing.

1.2 3DXRD and DCT 3D orientation mapping by monochromatic beam diffraction techniques is based on a quite different concept. The sample is illuminated with an extended (or line focused) monochromatic beam and rotated around an axis perpendicular to the beam. During the rotation over an extended angular range each grain will run through multiple diffraction alignments and give rise to diffracted beams. These diffracted beams are recorded on a high-resolution 2D detector system positioned closely behind the sample. For undeformed microstructures, the diffraction 'images' formed on the detector screen can be taken as approximations of 2D projections of the illuminated grain volumes. Polycrystal indexing algorithms classify these spots into sets belonging to the same grain and provide the average grain orientation and elastic strain tensors for each of the detected grains (Reischig 2008; Oddershede Schmidt, Poulsen, Sorensen, Wright and Reimers 2010). The 3D grain shape and position is calculated by means of algebraic tomographic reconstruction techniques using the 2D diffraction spots as projection input (Ludwig Reischig et al. 2009).

The illumination of the entire sample volume by an extended beam and detection of the diffraction spots on a high resolution detector system result in a tremendous gain in data acquisition speed: the sample needs to be scanned only around a single axis and micrometer spatial resolution is intrinsically provided by the detector system. Tomographic reconstruction methods or alternative analysis strategies (Suter, Hennessy, Xiao and Lienert 2006) provide access to the third dimension. For the case of undeformed monophase microstructures for which a description in terms of an average orientation per grain is appropriate, the outlined approach

provides optimum time resolution: sample volumes containing up to 500^3 voxels can be analysed within one hour - compared to 5 months for a 3D point scanning procedure at 10 Hz. In addition to the 3D orientation map, DCT offers complementary information in the form of a simultaneously acquired absorption tomogram.

The principal limitations of DCT and 3DXRD are the restriction to moderately deformed materials and the limited spatial resolution, determined by the detector system. Work is in progress to extend current 3DXRD methodology to deformed microstructures (West, Schmidt, Sorensen, Winther, Poulsen, Margulies and Gundlach 2009), higher spatial resolution (improved detectors) and to extend the capabilities of DCT in terms of characterization of sub-grain structures (King, Reischig, Martin, Fonseca, Preuss and Ludwig, this conference).

In this paper we describe an acquisition geometry optimized for spatial, strain and time resolution for (line focused) monochromatic beam diffraction experiments at X-ray energies below 25 keV (Section 2). We present first experimental results obtained in this configuration in Section3. The potential of this modified approach is compared to polychromatic diffraction experiments in Section 4.

2. ACQUISITION GEOMETRIES

In a 3DXRD grain mapping experiment one has the choice between different illumination modes ('box' beam versus 'line' beam versus 'pencil' beam) and detector positions. So far, data have been collected in different illumination modes - but using exclusively the forward scattering geometry with the detector normal to the incident beam direction. As already stated earlier, the choice of this 'standard' acquisition geometry is natural for experiments with highly absorbing samples, where the use of high X-ray energies is mandatory.

At energies below ~ 25keV, there is an additional degree of freedom in the sense that one can detect diffracted signals at higher and higher diffraction angles. However, due to the limited size of the detector, one can no longer intercept the full diffraction cone and one has to work with a translation and/or rotation offset of the detector in order to capture beams diffracted at high angles. The acquisition geometry is now a function of several parameters: the illumination mode and the detector position with respect to the incident beam / sample. For most high resolution X-ray imaging detector systems an additional consideration comes into play: beams impinging on the detector far from normal incidence can not be imaged with optimum resolution due to the broadening caused by the finite thickness of the scintillator screens. In order to preserve optimum spatial resolution, the detector is usually positioned perpendicular to the diffracted beam. For the rest of the discussion we will focus on detector configurations with close to normal incidence. (However, when working at low X-ray energies the thickness of the scintillator screens can be reduced to the micrometer range and the above mentioned parallax effects become less critical. Some systematic work is required to characterize the image degradation at oblique angles of incidence and its possible correction by deconvolution techniques.)

The linear polarization of the synchrotron beam implies a $\cos^2 2\theta$ dependence of the diffracted intensity when working in the horizontal diffraction plane (i.e. the standard 3DXRD / DCT geometry with the sample rotating around a vertical axis). For detection of diffracted beams at

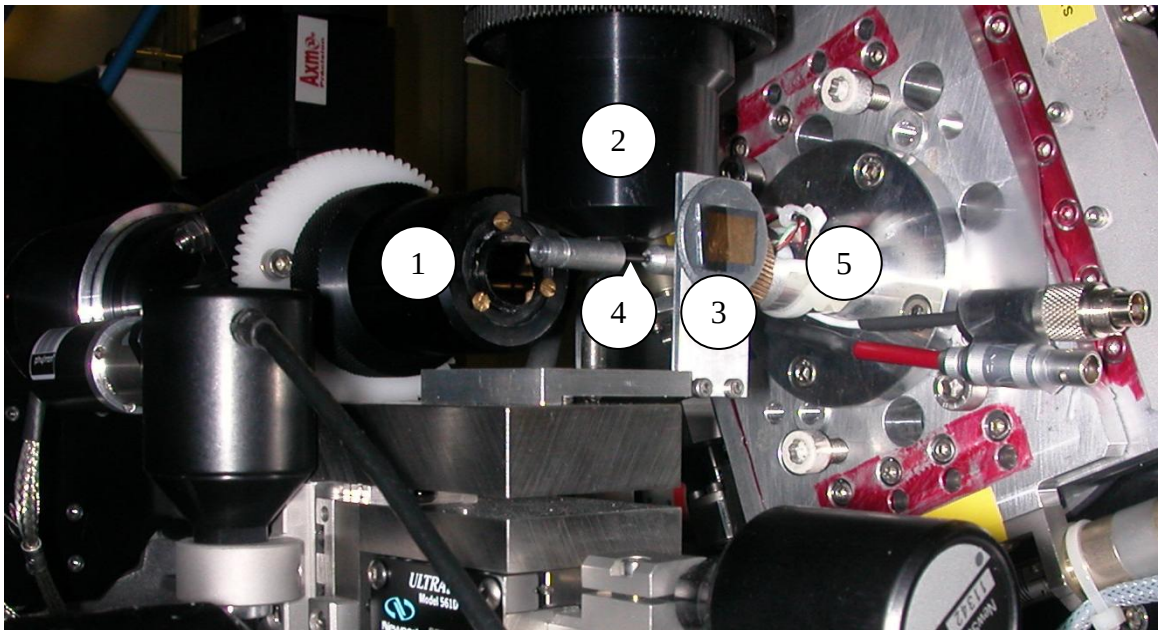

Figure 1: *Picture of the experimental setup. The beam arrives from the right and the direct and forward diffracted beams are recorded on a high-resolution detector system (1). Beams diffracted at angles close to 90° are recorded on a second high-resolution detector system (2), positioned vertically above the sample. The beam profile can be changed from extended (2D) to line beam (1D) by insertion and vertical scanning of a micro slit (3) aligned parallel to the rotation axis. The sample (4) is mounted in a miniature compression rig (5), positioned on the axis of a precision rotation table in horizontal configuration.*

angles close to 90° it is mandatory to switch to the vertical diffraction plane. This requires a modification of the setup with the rotation of the sample around a horizontal axis and an additional detector system positioned vertically above the sample (Fig. 1).

For the case of box beam illumination where the "thickness" of the grain is encrypted in the local intensity of the projection images, there is no obvious advantage in collecting data at a high rotation offset angle of the detector: similar projections of the grain would be acquired in standard geometry after rotation of the sample by the offset angle. There is, however, a subtle difference: the distortion of the projection images due to elastic strain and orientation gradients inside the grain will be more pronounced at high diffraction angles. Data acquired at small and high angles are therefore non-redundant and both can be used to confine the solution of iterative algorithms aiming at the reconstruction of the local orientation and strain distributions inside deformed grains (Reischig, Ludwig, King, Baumbach).

2.1 Line beam scanning procedure The main advantage of the modified acquisition geometry becomes apparent when reducing the dimensionality of the illumination from 2D (box beam) to 1D (horizontal line beam) mode. Only a single 'slice' of the grain is illuminated at a time and by scanning the line beam across the sample, a 3D grain volume can be assembled by stacking the individual layers. A similar line beam scanning procedure has already been used in standard geometry for the in-situ observation of an individual grain during the recrystallization process (Schmidt, Nielsen, Gundlach, Margulies, Huang and Juul Jensen 2004). The modified acquisition geometry (horizontal rotation axis and vertical detector) offers the potential to go beyond this achievement. First, beams diffracted close to 90° can now be recorded in optimum conditions benefiting from close to normal incidence and small geometric distortion since the

illuminated layer and the detector plane are parallel to each other. Improved, isotropic in-plane spatial resolution is a direct consequence. Second, the analysis of the Friedel pair of the diffraction spot gives access to the direction of the diffracted beam.[1] Knowing the line beam position, one can therefore back-project the diffraction images to their corresponding location in the 3D sample volume with high precision. A three-dimensional reconstruction of an individual grain obtained with this procedure is presented in section 3. Note that this kind of 3D reconstruction is obtained by simple stacking of slices and does not involve the solution of an inverse, tomographic problem.

The outlined scanning procedure offers potential for the reconstruction of 3D grain shapes in sample volumes with hundreds of grains from a limited angular range (e.g. 0-10° and 180-190°). The close spacing of reflections at high diffraction angles compensates for the fact that the vertical detector intercepts only a small segment of the diffraction cone. If grain orientations and positions are known from an indexing scan, the number of line scans can be reduced to a minimum by scanning only those omega and z positions at which reflections from the individual grains can be observed. The accuracy of the grain maps produced with this simple projection approach depends critically on the deformation state of the grains: the presence of orientation and/or strain gradients inside the grain can lead to severe distortion of the diffraction spots and hence erroneous 3D reconstructions.

2.2 Backscattering geometry Optimum sensitivity for the measurement of global elastic strain tensors is expected in backscattering geometry at two-theta values close to 180°. This requires a special detector with a central hole for the incoming beam. For the case of a classical X-ray imaging detector system composed of a scintillating screen, mirror and visible light optic coupling to a CCD camera, this can be easily achieved by modifying the mirror and scintillator screen. Due to the strong decay of the integrated intensity for high order reflections, detection in backscatter geometry is most efficient at low X-ray energies. This acquisition geometry may therefore imply restrictions to the sample thickness, especially for the case of highly absorbing materials. A first test experiment performed on a 500 µm diameter Al sample at an energy of 17.6 keV confirms detectability of backscattered diffraction spots (the quantitative analysis of these data is still outstanding). Ideally, the strain tensor error figures obtained from measurements of the same sample in different acquisition geometries should be analysed in order to evaluate different acquisition geometries against each other.

3. EXPERIMENTAL RESULTS

The experiments were performed at endstation ID18F of the European Synchrotron Radiation Facility (ESRF). The high beta straight section of this beamline is equipped with three single harmonic undulators, tuned to 14.4 keV. The beam is monochromatized by a liquid nitrogen cooled Si 111 double crystal monochromator At the sample position (60 m from the source) a beam has a size of 1200 (H) x 800 (V) µm and an integral flux of $5 \cdot 10^{13}$ photons/s. Two high resolution imaging detector systems were used, one centred on the direct beam (forward scattering geometry) and one vertically above the sample to record beams diffracted at angles

[1] Since the line beam and rotation axis are parallel to each other, the illuminated sample volume changes during rotation of the sample. Friedel pairs of diffraction spots originating from a given grain cross section can be observed after translation of the line beam to its symmetry position on the opposite side of the rotation axis, combined with 180° rotation of the sample.

close to 90°. Both systems were equipped with a transparent luminescent screen (Martin and Koch 2006), visible light optics and a FRELON camera (Labiche, Mathon, Pascarelli, Newton, Ferre, Curfs, Vaughan, Homs and Carreiras 2007). The effective pixel size on the forward scattering and vertical detector was 3.5 µm and 1.4 µm, respectively.

A cylindrical sample with 500 µm diameter and 1.5 mm height was prepared from a fully recrystallized Al 3% Li alloy with average grain size of 150 µm. The sample was mounted in a miniature compression rig equipped with a 500 N load cell. The load frame of the compression rig consists of a 1.5 mm outer diameter quartz capillary with 250 µm wall thickness.

A horizontal slit of 5 µm height, fabricated by micro-lithography techniques[2], could be inserted at a distance of about 20 mm upstream of the sample position. The slit was mounted on a motorized translation stage and could be scanned in vertical direction in order to illuminate horizontal cross-sections of the sample. Before the beginning of the experiment, the micro-slit was aligned parallel to the rotation axis with the help of a manual tilt adjustment.

3.1 Conventional DCT 3600 integrated projection images were recorded while rotating the sample continuously over 360° around the horizontal axis, aligned perpendicular to the X-ray beam. The diffraction data were analysed on-line, following the standard DCT processing route. Figure 2 shows a cross section through the preliminary (on-line) reconstruction of the 3D sample volume. Based on this map, a grain close to the center of the sample was selected for further characterization of local orientation and elastic strain fields by means of the line beam scanning procedure.

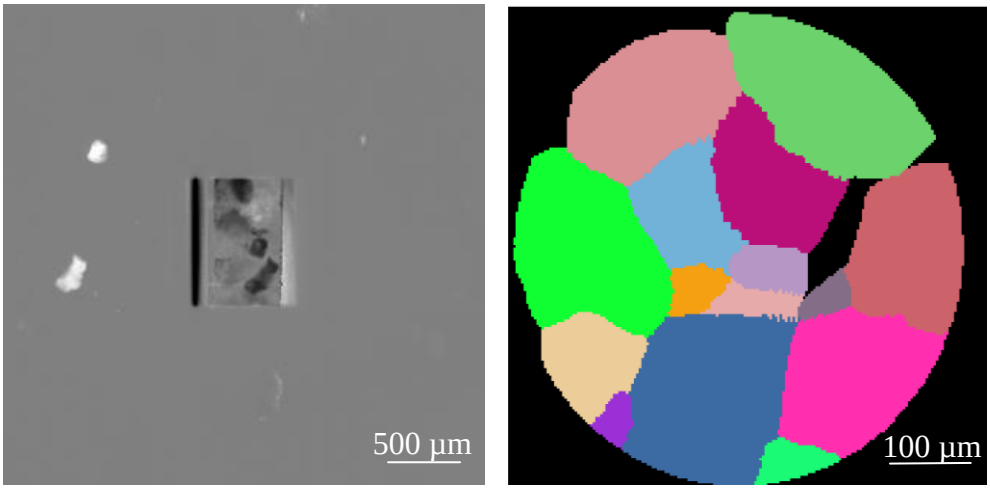

Figure 2. a) Background corrected projection image on the forward scattering detector. Slice through the 3D grain map produced during the experiment using the standard DCT processing route.

Line beam scanning procedure While processing the initial DCT reconstruction (~12 hours), an arbitrary reflection of one of the grains was selected and two line beam scans were performed for the reflection and its Friedel pair. Each image was integrated over a ω range of 0.44°, covering the entire width of the reflection curve. A total of 121 images were taken at vertical intervals of 2.8 µm for both reflections. Figure 3 shows the central slice through the grain for both reflections.

2 The microslit was fabricated at the Laboratory for Micro- and Nanotechnology (Paul Scherrer Institute). The slit itself consists of a 5 x 35 x 10000 µm silicon structure on top of a thin substrate (both ~transparent to X-rays), embedded in a 30µm gold layer.

Once the orientations and positions of the grains were known from the DCT reconstruction, a similar scanning procedure was set up for one selected bulk grain. First, the omega rotation positions for which reflections from this grain can be observed on the vertical detector were calculated. Knowing the shape and position of the grain, line scan macros were generated for 12 Friedel pairs. The start and end positions of the vertical scans were adjusted automatically to the position occupied by the grain at the corresponding omega rotation angles. The quantitative analysis of these data aiming at the reconstruction of the local orientation and strain distributions is in progress and will be reported elsewhere.

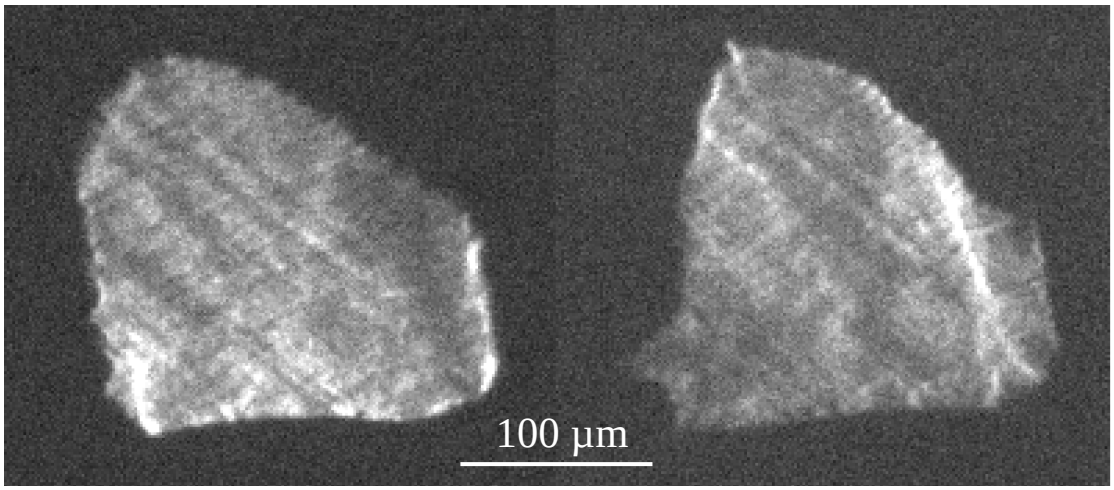

Figure 3: Friedel pair of section topographs through the central slice of the grain. Both images are integrated over 0.44° in ω rotation angle. The asymmetry of the spots is caused by orientation gradients within the grain.

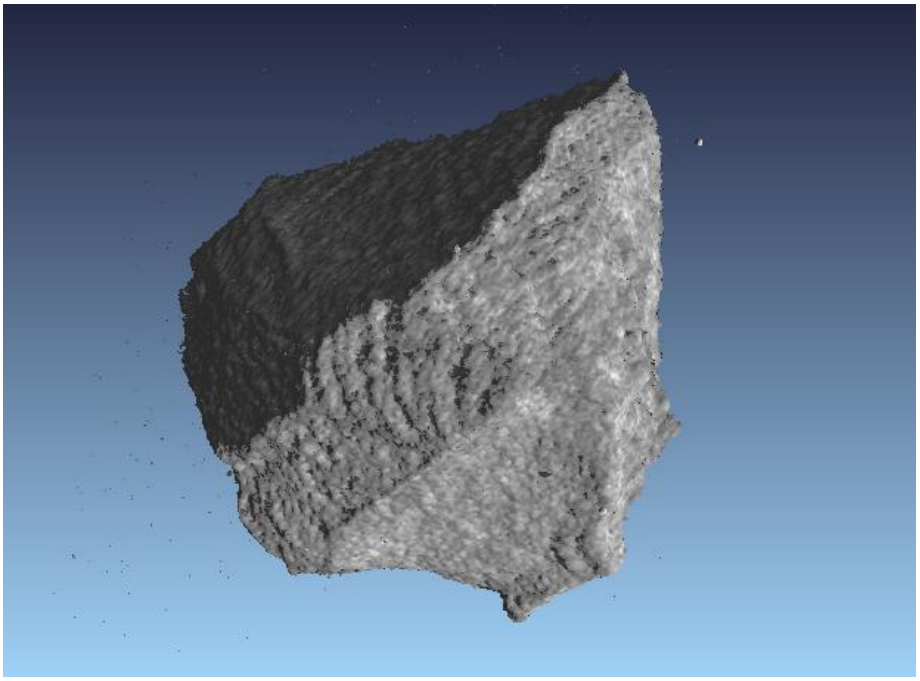

Figure 4: Rendering of the 3D grain volume obtained from stacking individual slices from the reflection shown in Figure 3b.

4. DISCUSSION

The acquisition of the diffraction spots at diffraction angles close to 90° and at X-ray energies below 20 keV apparently improves the spatial resolution achievable with classical 3DXRD line beam scans in forward scattering geometry. However, closer inspection of the Friedel pair of section topographs in Figure 3 clearly reveals the limitations of any grain reconstruction approach assimilating the diffraction images as parallel projections of the 3D grain volume. The presence of different orientations and strain gradients inside the grain leads to a pronounced asymmetry of the Friedel pair images: the grain contours are different and regions of high intensity in one image correspond to regions of low intensity in the other and vice versa. This behaviour is a clear signature of 'orientation contrast', which in turn can be identified as one of the main sources for discrepancies in the intensity distributions of Friedel pairs, observed in earlier experiments One can conclude, that the accuracy of any reconstruction approach which does not explicitly take the local degrees of freedom in orientation and elastic strain state of the material into account, will be limited by this effect.

Let D denote the distance between sample and detector and ψ be the angle between the diffracted beam and the detector surface normal. A given change $\Delta\eta$ in the direction of the diffracted beam will result in displacements of $u=D\cdot\tan\psi\cdot\Delta\eta$ and $u=(D/\cos\psi)\cdot\Delta\eta$ on the forward and vertical detector screens, respectively. The vertical detector configuration provides maximum sensitivity for variations in $\Delta\eta$ and this configuration should therefore be beneficial for any approach aiming at the reconstruction of small variations in local strain and orientation distributions.

In the same line of arguments, 3D grain reconstructions based on simple back projection (i.e. the current DCT approach) should preferentially be performed from data acquired on the forward scattering detector which are less affected by the image distortions discussed above.

Compared to the conventional geometry with a vertical rotation axis, one has to deal with the possible degradation of the mechanical performance (sphere of confusion) when placing the rotation stage in horizontal position, especially if any additional sample environment is required. It should be noted that there is a possibility to benefit from improved spatial and strain resolution in conventional geometry, too. Beams diffracted in the vertical direction and originating from the upper part of a sample can still be detected on the vertical detector. However, the corresponding Friedel pair is not accessible in this case (vertically below the sample), and one has to use tracking techniques or a three-dimensional detector in order to determine the diffraction vectors of the corresponding spots.

One of the main issues which needs to be addressed when considering reconstruction of local distributions from monochromatic beam diffraction data is the question if, and under which conditions, monochromatic micro-diffraction techniques can compete with established concepts of polychromatic diffraction techniques like DAXM in terms of time and spatial resolution. In the next paragraphs, tentative estimates of scanning times will be given for two different characterization tasks:

(1) the full 3D volume below a surface area of 100 x 100 elements has to be characterized (about 1000 grains, assuming an average grain size of 10x10x10 elements)

(2) a specific grain (dimensions 10x10x10 elements) of known position and orientation needs to be characterized with optimum time resolution.

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We will first consider the special case where characterization of the microstructure in terms the 3D grain shape and *global* (grain average) values for the orientation and elastic strain are appropriate (e.g. studies of grain growth) and then consider the more general (and challenging) case, where characterization in terms of *local* values inside the sample/grain volume is required (deformation studies).

4.1 Characterization of undeformed grains in terms of *global* orientation and elastic strain values

- In polychromatic scanning mode the investigation of the volume below a 100x100 surface area will require approximately 400.000 s (assuming that the wire needs to be scanned over a distance of about 400 units in order to cover a solid angle of 90 degrees on the detector). By doing so, the local orientation and the deviatoric components of the elastic strain tensor are acquired simultaneously from the material situated below the 100x100 unit footprint area. The actual achievable characterization depth is a function of the absorption of the material and the maximum energy in the spectrum of the incoming beam. We will assume that the diffracted signals can be recovered from sample depths of order of 100 units (10 'grains' in the depth direction).

  If the orientation and position of a specific grain are known, the wire scanning procedure can probably be restricted to regions of interest and acquisition times of order of 40 s per grain seem possible, provided that access to the deviatoric components of the strain tensor is sufficient.

- In 2D monochromatic scanning mode (e.g. DCT type of acquisition), a full scan with 3600 images at 10Hz will require 360 s. A sample volume of approximately 400x400x400 voxels, corresponding to (100x100x100 units, or 1000 'grains') can be characterized in terms of their *average* orientation and elastic strain tensors per grain in this case.

  If the orientation of a specific grain is known from a previous scan, one can in principle determine its average orientation and strain tensor from a minimum of 3 Friedel pairs of diffraction spots. However, reliable 3D shape reconstructions require about 10 projections upwards (~2 s, neglecting finite positioning times of the rotation stage).

As a first conclusion we can state that DCT / 3DXRD are superior in time resolution for cases in which a description in terms of *average* quantities is sufficient (e.g observation of grain growth). However, the situation becomes less clear when local (sub-grain) information is required (e.g. deformation studies).

4.2 Characterization of moderately deformed grains in terms of *local* orientation and strain values For the moment it is still unclear up to which degree of plastic deformation one can extract local orientation *and* strain information from extended (2D), monochromatic beam diffraction data (see King et al., this conference). The change to the horizontal line beam illumination mode is a straightforward way to reduce the convolution of the diffraction data and hence to increase the chances for successful reconstruction of these local variables. For the rest of the discussion we assume that iterative (or other) algorithms can recover this information from data acquired in this geometry.

However, replacing the one-dimensional (ω) by a two-dimensional (ω,z) scanning procedure, the speed of data acquisition drops and one has to question if, and in which conditions, monochromatic beam diffraction can still compete with polychromatic diffraction experiments in terms of time resolution in these situations.

If no a priori information on the sample is available, the line beam scan has to be repeated for all the rotation angles. Changing from 2D to 1D illumination mode will therefore typically result in a 100 times increase of the scanning times (here we assume that the additional scan over the vertical sample dimension comprises 100 steps and that focusing of the beam in one dimension will compensate for the loss of the diffraction signal at the detector). In monochromatic beam the full scanning procedure would require 360*100 s (10 h) for ~1000 grains.

If grain orientations and positions are known, and the task is to determine the local variables in one specific grain, the factor 100 increase of the total scanning time can be reduced considerably, since only a minimum of three Friedel pairs of reflections have to be acquired (each spreading over, say 2 degrees). In addition, the range of the vertical scan can be reduced to the dimensions of the grain (10 units). At ω steps of 0.1° this amounts to a minimum of 3*2*20*10 images or 120 s for one grain - compared to 40 s in polychromatic diffraction, assuming a known grain position and orientation as well.

In conclusion, the general trend seems to be that DAXM can provide faster local characterization for an individual grain of known position and orientation, whereas the monochromatic beam line scanning procedure seems to provide an advantage when characterization of large sample volumes with hundred and more grains is required.

4.3 Ultimate spatial resolution The ultimate spatial resolution of polychromatic diffraction experiments is determined by the minimum achievable spot size, positioning accuracy, vibration and long term stability of the instrument. The current state of the art is of order of 200 nm. By contrast, the resolution of the monochromatic diffraction experiments described in this paper is limited by the spatial resolution of the X-ray imaging detector system and the achievable structure size of the beam defining apertures. The state of the art in terms of FWHM of detector point spread functions is about 500nm (with effective pixel size of order of 200 nm). Grouping voxels into elements of 4x4x4 voxels finally results in an ultimate spatial resolution of about 1 µm with current concepts and technology.

5. CONCLUSIONS

The line beam acquisition procedure and geometry presented in this paper enables high spatial resolution reconstruction of 3D grain shapes by means of a one-dimensional scan of a beam-defining aperture. If grain orientations are known from an indexing scan, the acquisition of one line scan per grain can replace the tomographic reconstruction from a set of projections taken at different rotation angles.

Acquiring line scans for different reflections of the same grain and using iterative reconstruction procedures, the reconstruction of local orientation and elastic strain tensors within individual grains can be envisaged. The expected scanning times for the polychromatic and monochromatic diffraction approach may differ by one order of magnitude in both directions and depend on the number of grains to be analysed. Both approaches have complementary strengths and drawbacks. In particular, monochromatic beam diffraction can be readily combined with 3D X-ray imaging (absorption and phase contrast tomography on the same instrument) and provides access to the full elastic strain tensor at the expense of spatial resolution (1µm) and limited capability for studying strongly deformed or multi-phase materials. Polychromatic micro-diffraction on the other hand can handle multiphase and heavily deformed materials, but is blind to the absorption microstructure and requires switching between monochromatic and polychromatic scanning, if the hydrostatic component of the strain tensor needs to be determined.

Reconstruction of local distributions from extended (2D), monochromatic beam diffraction data would result in a tremendous gain in acquisition speeds and efforts in this direction need to be pursued. The horizontal line beam scanning approach presented in this paper can be used to verify results obtained from extended beam diffraction data and should help establishing appropriate reconstruction algorithms for the general 2D case. The use of diffraction images acquired at high scattering angles can be expected to improve the sensitivity for weak elastic strain and orientation changes.

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